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(71) Applicant (*for all designated States except US*): **W. & J. LEIGH & CO.,** [GB/GB]; Tower Works, Kestor Street, Bolton BL2 2AL (GB).

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(72) Inventors; and
(75) Inventors/Applicants (*for US only*): **FARRELL, Peter, John** [GB/GB]; Tower Works, Kestor Street, Bolton BL2 2AL (GB). **GREEN, John, Darryl** [GB/GB]; Tower Works, Kestor Street, Bolton BL2 2AL (GB).

(74) Agents: **GOODWIN, Mark** et al.; Wilson, Gunn M'Caw, 41-51 Royal Exchange, Cross Street, Manchester M2 7BD (GB).

(54) Title: COATING COMPOSITIONS

(57) Abstract: An intumescent coating composition in a solid state comprises from 20% to 80% by volume of a polymer resin system together with from 15% to 75% by volume of intumescent ingredients which, under the action of sufficient heat, are a source for acid, carbon and gas, the composition comprising from 25% to 85% of the acid source by weight of the intumescent ingredients content of the intumescent coating composition, from 1% to 50% of the carbon source by weight of the intumescent ingredients content of the intumescent coating composition and from 1% to 50% of the gas source by weight of the intumescent ingredients content of the intumescent coating composition.



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Coating Compositions

The present invention relates to intumescent powder compositions which can be applied to various substrates so as to form thereupon an intumescent coating. A major application of the compositions of the invention is in the coating of steel, and in particular structural steel used in the manufacturing of buildings and also steel plate and steel sheeting known as "strip".

Intumescent coating technology has been commercially available for some time based on liquid resin technology or resin solutions. Intumescent coatings have been developed to provide a more aesthetically pleasing alternative to the previously preferred boarding materials used for thermal protection and/or fire retardation.

Intumescent coatings are designed to "swell up" in the event of a fire, a reaction initiated at a given temperature, forming an insulating char or foam which shields the underlying structural components from the heat thereby delaying the time taken for the structural components to reach their critical failure temperature (CFT). The CFT is the temperature at which the structural integrity of steel components is compromised. If the aforementioned components form part of the structure of a building, then this may lead to collapse of the building.

The delay in reaching the CFT enables safe evacuation of the building whilst increasing the time available to fire fighting teams to extinguish the fire thereby greatly reducing the risk of the building collapsing.

The intumescent coatings currently available are applied in liquid form via standard airless spray equipment, a standard technique for industrial paint application.

The thickness of the dry intumescent coatings ranges from 250 microns (3-5

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times thicker than conventional paint thickness) up to several millimetres thick depending upon the type and size of the structural components and the duration of protection required in the event of a fire.

The currently available intumescent coatings are accompanied by long drying times particularly at the aforesaid elevated thickness. It is not uncommon to allow several days between the application of the intumescent coating and the handling of the coated structural component in order to prevent damage occurring to the coating.

Accordingly, it is impractical to apply the said intumescent coatings to structural components and transport these structural components to site the same day. Additionally the application of the said intumescent coatings "on site" causes delays in the construction process which may result in significantly increased financial burdens.

Recently, faster drying intumescent coatings have been made available. However, for the most part, the drying time remains in excess of 1-2 days.

Therefore, it is still not possible to apply liquid intumescent coatings to structural components and transport these structural components to site the same day when the said intumescent coatings have been applied "in shop".

Powder coatings have been commercially available for some years and are currently one of the fastest growing areas of protective coatings.

Advantageously, when compared to wet paint, powder coatings require no volatile organic compounds as they are 100% solid and any material losses are minimised due to the electrostatic deposition and the potential recycling capabilities of the powder. They also have excellent film appearance and strength, facilitate even

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application of thickness and provide good coverage of difficult-to-reach places.

However, methodology restricts the use of powder coatings to relatively small or thin substrates and also limits the thickness that can be applied due to electrostatic shielding and/or back-ionisation. These difficulties have up until now discouraged industry from developing an intumescent powder as it needs to be applied at much higher thickness than typical powder technology allows.

Electrostatic spray application is the most common method for the application of powder coatings whereby charged powder particles are sprayed onto earthed substrates where binding occurs via opposite charge effects. Curing is achieved by oven baking. Due to the nature of the application and curing, the size and type substrates that can be sprayed in this way is limited. Film thickness of up to 200 microns may be achieved by this method.

Film formation and curing requires a temperature typically in the range of 140-200⁰C for 10-30 minutes in order for the powder coating to melt and flow thereby forming a complete cured film upon the substrate.

An alternative method of application is via a fluidised bed, whereby the substrate is pre-heated in an oven to or above the required powder flow temperature and this is then immersed in an atmosphere of free flowing powder particles. These particles then melt and flow upon contact with the hot substrate thereby forming a complete film of typically 100-1000 microns thick.

Unfortunately, the aforesaid methods alone do not lend themselves well to the coating of large substrates. Moreover, the aforesaid methods have not previously been applied to the application of intumescent coating compositions.

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The structural components to which intumescent coatings are applied may be constructed of steel and can be up to several metres in length with a steel thickness ranging from about 1mm to 50mm. This invention offers an alternative approach to the intumescent coating of structural steel, steel plate and "strip".

The present invention seeks to provide a solid intumescent coating composition and a method of producing and applying that composition to substrates of the aforesaid dimensions whereby coating thickness required for a minimum of thirty minutes fire protection can be applied in a commercially viable number of coating operations.

According to a first aspect of the present invention there is provided an intumescent coating composition in a solid state comprising from 20% to 80% by volume of a polymer resin system together with from 15% to 75% by volume of intumescent ingredients which, under the action of sufficient heat, are a source for acid, carbon and gas, the composition comprising from 25% to 85% of the acid source by weight of the intumescent ingredients content of the intumescent coating composition, from 1% to 50% of the carbon source by weight of the intumescent ingredients content of the intumescent coating composition and from 1% to 50% of the gas source by weight of the intumescent ingredients content of the intumescent coating composition.

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The intumescent coating composition of the invention may be applied to structural steel and the like in a cost-effective, environmentally friendly coating operation.

The binder of the present invention may comprise a thermoplastic resin system or a thermosetting resin system. Preferably, the binder of the present invention is a thermoplastic resin system or a thermoplastic/thermoset hybrid.

Suitable thermosetting components include epoxy, polyester, such as TGIC polyester, modified epoxy (modified with, for example, acrylic, polyester), acrylic urethane.

The resin system should fluidise at temperatures below the initiation temperature of the intumescent reaction and at temperatures low enough to facilitate application resulting in the application of a continuous film up on the structural components. Accordingly, the resin system ideally fluidises below 250°C and preferably below 200°C. More preferably the resin system fluidises between 90 and 180°C and most preferably between 120°C and 150°C.

It has been found that in order for the coating compositions of the present invention to perform at an optimal level, the resin system should predominantly consist of a thermoplastic resin.

Preferably, the resin system of the present invention shall comprise at least 65%, and ideally at least 75%, of thermoplastic resin, by weight of the resin system as a whole.

Powder coating compositions comprising predominantly thermoplastic resin systems are desirable in that such materials are capable of being applied at much

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higher film thicknesses than resin systems comprising solely thermoset polymers. Furthermore, thermoplastic resins may be re-coated indefinitely. Therefore film coatings, of 1mm or more may be achieved using intumescent compositions having a resin system consisting largely of thermoplastic resins. Large coating thicknesses are necessary to provide sufficient insulation to the substrate during a fire. The ease of coating also facilitates repair and maintenance of the coating on the coated substrate.

Suitable thermoplastic resins include any of the following either alone or in combination:- polyethylene (low density and/or linear low density), ethylene/vinyl acetate copolymers, ethylene/acrylic acid copolymers, polypropylene or thermoplastic epoxies (i.e. polyetheramines). The thermoplastic nature of the resin means that substantial intumescence is obtained providing excellent insulation to the coated substrate e.g. steel, during a fire.

One most preferred thermoplastic resin would comprise at least one polyolefin (such as polyethylene), the film of which is quite tough, but relatively soft and flexible. Such a film could be useful in cable coatings and "soft feel" coatings.

Another most preferred thermoplastic resin would comprise at least one durable. Intumescent coatings comprising such resins would be suitable for structural steel.

The aforesaid thermoplastic components are particularly suitable as they are able to withstand the processing, high temperatures, pressure and shear exerted upon the coating compositions of the present invention both during their manufacture and in their use.

Preferably, the thermoplastic resin system shall comprise at least one BLOX

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resin (commercially available from Dow) comprising polyether-amines of low enough molecular weight to display thermoplastic properties whilst delivering the hardness, durability and adhesion to substrate qualities associated with a thermosetting resin component. The BLOX resin system fluidises at 120-150°C displaying sufficient flow to facilitate a good, smooth film upon heating.

The resin system preferably constitutes from 45% and 85% (by volume) of the intumescent coating composition.

The active ingredients consist of three essential components, an acid source, a carbon source and a spumific or gas source and preferably a nucleating agent.

Under the influence of heat (typically 200-300°C) the acid source, usually by decomposition, produces acid which then proceeds to react with other constituents in the coating. The acid reacts with the carbon source to yield large amounts of carbon as a product of the reaction. Meanwhile under the influence of heat (typically 200-400°C) the spumific, or gas source, usually by decomposition, produces gas which results in a bubbling or swelling of the coating which ultimately gives rise to an intumescent char or foam.

The intumescent coating composition of the present invention contains at least one acid source examples of which, which can be used alone or in combination, include ammonium polyphosphate, melamine phosphate, magnesium sulphate, boric acid and amine sulphates.

Preferably, the acid source will be ammonium polyphosphate.

The acid source preferably constitutes from 40% to 75% by weight of the intumescent ingredients content of the intumescent coating composition.

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The intumescent coating composition of the present invention contains at least one carbon source. Preferred carbon sources include any of the following either alone or in combination:- polyhydric alcohols such as pentaerythritol, di-pentaerythritol, tri-pentaerythritol and starch.

Preferably, the carbon source will be pentaerythritol, di-pentaerythritol or a blend of the two. The carbon source preferably constitutes from 5% to 35% by weight of the intumescent ingredients content of the intumescent coating composition.

The intumescent coating composition of the present invention contains at least one gas source, commonly known as a spumific, examples of which include any of the following either alone or in combination:- melamine, melamine phosphate, melamine isocyanurate, tris-(hydroxyethyl) isocyanurate (THEIC), ammonium polyphosphate and dicyandiamide.

Preferably, the spumific or gas source will comprise any of melamine, dicyanamide, ammonium polyphosphate and THEIC.

The gas source preferably constitutes from 5% to 35% by weight of the intumescent ingredients content of the intumescent coating composition.

The intumescent coating composition of the present invention preferably contains at least one nucleating agent, examples of which include any of the following either alone or in combination:- titanium dioxide, silicon derivatives such as fumed silica, mica, and Bentonite clays.

Preferably, the nucleating agent of the present invention will be titanium dioxide.

The nucleating agent preferably constitutes from 1% to 30% by weight of the

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intumescent ingredient content of the intumescent coating composition and more preferably from 5% to 25% by weight of said content.

The active pigments content of the intumescent coating composition preferably constitute from 15% to 65%, by volume, of the intumescent coating composition of the present invention and preferably 20% to 50%.

Furthermore, additional additives may be included in the intumescent coating composition of the present invention. These include any of the following:- (i) one or

more flame retardant compounds which are added to aid char formation;

(ii) one or more metal salts which are added to improve char characteristics;

(iii) one or more silica/silicates which can act as nucleating agents; and/or

(iv) one or more fibre fillers which are used to strengthen char and prevent char degradation during a fire. Additionally, the fibre can improve rheological properties and prevent slumping during the heating process.

Suitable flame retardant compounds, which can be used alone or in combination include compounds containing either phosphorus, chlorine or isocyanurates, examples of which include ammonium polyphosphate, triphenyl phosphate, tricresyl phosphate, chlorinated paraffin, chlorinated rubber, tris-(2-chloroethyl) phosphate (TCEP), triglycidyl isocyanurate and tris-(2-hydroxyethyl) isocyanurate (THEIC). Further possible flame retardant compounds include aluminium hydroxide and/or magnesium hydroxide.

Preferably, the flame retardants comprise any of ammonium polyphosphate, aluminium hydroxide, THEIC and chlorinated paraffin.

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The flame retardants preferably constitute from 25% to 95% of the intumescent ingredients content and preferably from 40% to 85%.

Suitable metal salts include any of the following either alone or in combination:- zinc borate, zinc stannate, aluminium hydroxide, magnesium hydroxide and antimony oxide. The preferred metal salts include zinc borate and aluminium hydroxide.

The metal salts preferably constitute from 0 to 20%, more preferably 0% to 10%, and most preferably from 0% to 5%, of the intumescent ingredients of the coating composition.

Suitable silica / silicates include any of the following either alone or in combination:- fumed silica, carbonate free/containing silicates, Magnesium Silicate, Calcium Magnesium Silicate and Aluminium / Potassium Silicate.

The preferred Silica / Silicates include magnesium silicate and Aluminium / Potassium Silicate.

The silica / silicates preferably constitute from 0 to 20%, more preferably 0 to 15% and most preferably 0 to 10% of the intumescent ingredients of the coating composition.

Suitable fibre fillers include any of the following either alone or in combination :-silica fibre, ceramic fibre and proprietary intumescent fibre.

The preferred fibre fillers include silica fibre and proprietary intumescent fibre.

The fibre filler preferably constitutes from 0 to 20%, more preferably 0 to 15%, and most preferably 0 to 10% of the intumescent ingredients of the coating composition.

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Ideally the coating compositions of the invention can be applied in powder form to substrates up to several metres in length with a gauge thickness typically ranging from 5mm to 30mm whereby the film thickness provides adequate fire protection for at least 30 minutes.

According to a second aspect of the present invention there is provided the use of a composition according to the first aspect of the invention for coating a substrate.

The components of the composition are solid or semi-solid at room temperature. The components are preferably pre-blended to break down larger particles and render the pre-blend homogenous. The pre-blending is preferably carried out at room temperature. If the composition exceeds a certain temperature then it is likely that the intumescent reaction will be initiated. Extruders are then used to disperse the pigmentation within the resin system. This is achieved by the extruder heating the pre-ground powder to above the melting point of the resin and then dispersing the pigments in the resin by means of a series of screws. Care must be taken to ensure that the extruding temperature does not approach the temperature at which intumescence would occur. The extruded material is rolled into sheets, usually from 1 to 5mm thick, by means of a chilled roller situated at the end of the extruder. The composition may then be ground to produce particle sizes suitable for application, usually between 20-400 microns. If the composition contains thermoplastic resin systems the composition should ideally be cooled to below 20°C and preferably below 10°C to ease the grinding process.

Suitable methods for the application of the aforesaid compositions include any of:- electrostatic spray, a fluidised bed, hot/flame spray or powder slurry applications.

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If electrostatic spray application is used relatively high coating thicknesses (more than 1mm) can be achieved by pre-heating the substrate to a temperature that is hot enough to melt and fuse the applied powder upon contact e.g. 100-200°C. The thickness of powder can be increased for as long as the substrate temperature remains above this level. This is dependant upon the initial temperature and thickness of the substrate. Film formation (and curing if applicable) is effected between 100-200°C for a period of between 5-30 minutes, or by the use of near infra red heating for periods of between 1-20 minutes.

Advantageously, application via electrostatic spray enables application of the intumescent coating to occur without the need to turn the substrate.

Furthermore, electrostatic spray typically gives rise to a 98% transfer efficiency compared with 50% when spraying wet paint.

Application using a fluidised bed is suitable for film thickness of up to and above 1mm. No heating is required after the coating has been applied providing that acceptable flow and film formation has occurred during the process.

Application via hot/flame spray is suitable for unlimited thicknesses in a multi-layer system. This application process requires the particles to be melted by means of a propane flame, and applied onto the substrate to form a film, which then cools and hardens.

Application may alternatively be by the use of a "powder slurry" which involves dispersion of the powder coating in a liquid medium, usually water, along with other miscellaneous additives such as thickeners / thixotropic agents, surfactants, dispersing and wetting agents. This method allows the powder to be applied as a

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“slurry” via traditional spraying methods, for example, airless, conventional, airless-air assisted or HVLP (High volume low pressure). Again significantly high thicknesses, i.e. above 1mm, can be applied in one coat provided the slurry has been produced with the necessary levels of thixotropising agent or agents. After allowing the liquid media to evaporate the powder is then heated in like manner to the previously described methods.

The inclusion of a thermoplastic resin either as the whole or part of the binder in the composition allows multi-coat applications with good inter-coat adhesion if greater film thickness is required.

Film formation (and curing if applicable) temperatures must not exceed the temperature at which the intumescent reaction is initiated.

Advantageously, the application of intumescent coating compositions in the powder form opens up new possibilities for the utility of intumescent coatings.

For example, thin strip steel is typically provided in rolls. The drying time of intumescent coatings currently available does not lend itself to the production of rolls of thin strip steel.

However, the composition of the present invention is ideally suited for the aforesaid application.

In order that the present invention may be more readily understood specific examples thereof are set out below.

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Example Formulation 1

Component	Approximate % by weight
Nucleating agent ¹	6
Acid Source ²	17
Carbon Source ³	6
Gas Source ⁴	6
Thermoplastic polymer resin ⁵	65

Example Formulation 2

Component	Approximate % by weight
Nucleating agent ¹	5
Acid source ²	15
Carbon source ³	5
Gas source ⁴	5
Metal salt ⁶	2.5
Flame retardant ⁷	2.5
Thermoplastic polymer resin ⁵	65

Example Formulation 3

Component	Approximate % by weight
Nucleating agent ¹	5
Acid Source ²	20
Carbon Source ³	6.5
Metal Salt ⁶	2.5
Flame retardant ⁷	1.75
Thermoplastic polymer resin ⁵	62.5
Fibre ⁸	1.75

¹e.g. Titanium Dioxide, "Tioxide TR92" from Tioxide Europe

²e.g. Ammonium Polyphosphate, "FR CROS 486" from Budenheim Iberica Sa

³e.g. Pentaerythritol, "pentaerythritol Fine Grade" from Chance & Hunt Ltd

⁴e.g. Melamine, "Melamine grade 003" from DSM UK LTD

⁵e.g. Polyetheramine, "BLOX 2100" from Dow Chemicals

⁶e.g. Zinc Borate, "Zinc Borate 9506" from Joseph Storey & Co

⁷e.g. Aluminium Hydroxide, "Trihyde OL 104" from Omya UK

⁸ e.g. Silica Fibre Filler, "Klevowool S – Type 060" from Klevers Glass Fabrics

Test Results

Example formulations 1, 2 and 3 were manufactured by extrusion at 150°C , ground to below 400 µm particle size and applied by electrostatic spray onto

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preheated (150°C) steel I-sections (0.5M 152mmx152mmx37Kg, $H_p/A = 190$). Further heating to 160°C was then required to induce flow and film formation of the coatings. In order to gauge the fire resistance performance of the materials, film thicknesses of between 0.5 and 1mm were aimed for. Actual film thicknesses of 0.50mm (example 1), 0.59mm (example 2) and 0.68mm (example 3) were achieved.

The coated steel sections were tested in a 1M³ furnace following the general principals outlined in BS476 Part 20, 1987, using the cellulosic heating curve.

For example formulation 1, the steel section took 23 minutes to reach a mean temperature of 550°C (column failure temperature) and 27 minutes to reach a mean temperature of 620°C (beam failure temperature). Approximately 15-25mm of intumescent char/foam was generated.

For example formulation 2, the steel section took 25 minutes to reach 550°C and 29 minutes to reach 620°C. Approximately 15-25mm of char/foam was generated.

For example formulation 3, the steel section took 30 minutes to reach 550°C and 36 minutes to reach 620°C. Approximately 15-25mm of char/foam was generated.

It follows that if greater film thicknesses of coating are applied, longer times to reach the CFT (i.e. 550°C/620°C) will be achieved.

It is to be understood that the above described examples are by way of illustration only. Many modifications and variations are possible.

CLAIMS

1. An intumescent coating composition in a solid state comprising from 20% to 80% by volume of a polymer resin system together with from 15% to 75% by volume of intumescent ingredients which, under the action of sufficient heat, are a source for acid, carbon and gas, the composition comprising from 25% to 85% of the acid source by weight of the intumescent ingredients content of the intumescent coating composition, from 1% to 50% of the carbon source by weight of the intumescent ingredients content of the intumescent coating composition and from 1% to 50% of the gas source by weight of the intumescent ingredients content of the intumescent coating composition.
2. An intumescent coating composition according to claim 1, wherein the polymer resin system comprises at least 65% of a thermoplastic resin, by weight of the resin system as a whole.
3. An intumescent coating composition according to claim 1 or claim 2, wherein the polymer resin system comprising at least 75% of a thermoplastic resin, by weight of the resin system as a whole.
4. An intumescent coating composition according to any preceding claim, wherein the polymer resin system comprises at least one polyolefin.
5. An intumescent coating composition according to any preceding claim, wherein the polymer resin system comprises at least one polyetheramine.
6. An intumescent coating composition according to any preceding claim, wherein the polymer resin system fluidises below 250°C.

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7. An intumescent coating composition according to claim 6, wherein the polymer resin system fluidises below 200°C.
8. An intumescent coating composition according to claim 7, wherein the polymer resin system fluidises between 90°C and 180°C.
9. An intumescent coating composition according to claim 7, wherein the resin system fluidises between 120°C and 150°C.
10. An intumescent coating composition according to any preceding claim, wherein the polymer resin system constitutes from 45% to 85% by volume of the intumescent coating composition.
11. An intumescent coating composition according to any preceding claim, wherein the acid source comprises ammonium polyphosphate.
12. An intumescent coating composition according to any preceding claim, wherein the acid source constitutes from 40% to 75%, by weight, of the intumescent ingredients content of the intumescent coating composition.
13. An intumescent coating composition according to any preceding claim, wherein the carbon source comprises at least one polyhydric alcohol.
14. An intumescent coating composition according to any preceding claim, wherein the carbon source constitutes from 5% to 35%, by weight, of the intumescent ingredients content of the intumescent coating composition.
15. An intumescent coating composition according to any preceding claim, wherein the gas source comprises any of the following either alone or in combination:- melamine, dicyandiamide, ammonium polyphosphate or THEIC.

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16. An intumescent coating composition according to any preceding claim, wherein the gas source constitutes from 5% to 35%, by weight, of the intumescent ingredients content of the intumescent coating composition.
17. An intumescent coating composition according to any preceding claim, wherein the intumescent coating composition comprises at least one nucleating agent.
18. An intumescent coating composition according to claim 17, wherein the nucleating agent comprises titanium dioxide.
19. An intumescent coating composition according to claim 17 or claim 18, wherein the nucleating agent constitutes from 1% to 30% by weight, of the intumescent ingredients content of the intumescent coating composition.
20. An intumescent coating composition according to any of claims 17 to 19, wherein the nucleating agent constitutes from 5% to 25%, by weight, of the intumescent ingredients content of the intumescent coating composition.
21. An intumescent coating composition according to any preceding claim, wherein the composition further comprises at least one flame retardant.
22. An intumescent coating composition according to claim 21, wherein said flame retardant comprises any of the following either alone or in combination:- ammonium polyphosphate, THEIC and chlorinated paraffin.
23. An intumescent coating composition according to claim 21 or claim 22, wherein said flame retardant constitutes from 25% to 95% of the

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intumescent ingredients content of the intumescent coating composition.

24. An intumescent coating composition according to any of claims 21 to 23, wherein said flame retardant constitutes from 40% to 85% of the intumescent ingredients content of the intumescent coating composition.
25. An intumescent coating composition according to any preceding claim, applied to a substrate, wherein the thickness of the coating is at least 250 μ m.
26. The use of an intumescent coating composition as claimed in any preceding claim for coating a substrate.

INTERNATIONAL SEARCH REPORT

PCT7GB 02/02289

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C09D5/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 532 292 A (WAINWRIGHT ROBIN ET AL) 2 July 1996 (1996-07-02) column 3, line 26-35 column 4, line 18-64 column 5, line 38-57 ----	1,2,6-26
A	US 4 442 157 A (MARX HANS-NORBERT ET AL) 10 April 1984 (1984-04-10) -----	



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

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European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Miller, A

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